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OPTICAL ADHESIVE

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(54) [Title of the Invention]

Optical adhesive

[Claims of the invention]

[Claim 1] An adhesive containing at least one compound A selected from the group consisting of N-vinyl carbazoles and substituents thereof and at least one compound B selected

from the group consisting of N-vinyl pyrrolidones and substituents thereof and acryl amide N-substituents and having a refractive index (n_d) after curing of at least 1.60.

[Claim 2] The adhesive of Claim 1 wherein the N-vinyl carbzole substituent is 3-nitro-N-vinyl carbazole, 3-amino-N-vinyl carbazole, 3-methyl amino-N-vinyl carbazole, 1,2,3,4-tetrahydro-N-vinyl carbazole, or 3,6-dibrom-N-vinyl carbazole; the N-vinyl pyrrolidone substitutent is N-vinyl-3-methyl pyrrolidone, N-vinyl-5-methyl pyrrolidone, N-vinyl-3-benzyl pyrrolidone, or N-vinyl-3,3,5-trimethyl pyrrolidone; the acryl amide N-substituent is N-methyl acryl amide, N-ethyl acryl amide, N-n-propyl acryl amide, N-n-butyl acryl amide, N-allyl acryl amide, N,N-dimethyl acryl amide, N,N-diethyl acryl amide, N,N-dimethyl amino propyl acryl amide or acryloyl morpholine.

[Claim 3] The adhesive of Claim 1 or Claim 2 characterized by the fact that a polymerization initiator is included.

[Claim 4] The adhesive of one of Claims 1~3 wherein 30~97 wt% of compound A is included.

[Claim 5] The adhesive of Claim 4 wherein 30~65 wt% of compound A is included.

[Detailed description of the invention]

[Field of industrial application]

The present invention pertains to a transparent adhesive used for adhesion of optical components, and the invention further pertains to an optical adhesive having a very high refractive index after curing.

[Prior art]

In the past, Canadian balsam, acrylic base adhesives and epoxy base adhesives are mainly used for bonding of optical components. Furthermore, in order to prevent loss due to reflection of light at the boundary between the optical component and the adhesive based on the difference in refractive index, adhesives where the refractive index of the adhesive is matched to the refractive index of the optical components have been proposed in recent years.

For the above-mentioned adhesives, for example, adhesives containing glycerol-modified bisphenol A di(meth)acrylate and glycerol-modified fluorine-containing bisphenol A di(meth)acrylate as essential ingredients can be mentioned, and adhesives where the refractive index (n_d) can be adjusted within the range of 1.45 to 1.59 are being marketed (Polymer Preprints, Japan vol. 38, No. 5(1989), p.1284, p.1304).

[Problems to be solved by the invention]

However, an optical material or optical element having a refractive index (n_d) of at least 1.60 is required in the design of optical waveguides and optical systems wherein many optical lenses are used in combination. When the adhesives of the prior art with low refractive indexes are used for bonding the above-mentioned optical components, losses due to the difference in the refractive index between the optical components and the adhesive pose a problem as explained above.

The purpose of the present invention is to eliminate the above-mentioned problem of the prior art and to produce a high-quality optical adhesive having a high refractive index.

[Means to solve the problem]

Thus, the present invention pertains to an adhesive containing at least one compound A selected from the group consisting of N-vinyl carbazoles and substituents thereof and at least one

compound B selected from the group consisting of N-vinyl pyrrolidones and substituents thereof and an acryl amide N-substituent and having a refractive index (n_d) after curing of at least 1.60.

Compound A of the present invention is a polymeric material having a high refractive index and remains a solid crystal at room temperature.

Compound B of the present invention is a polymeric material as in the case of compound A and has high adhesion with optical components such as glass based on hydrophilic properties. Furthermore, the compound is capable of dissolving compound A at high concentrations and has excellent copolymeric properties with that compound; thus, a material capable of very high transparency after curing is achieved.

Therefore, when compound A and compound B of the present invention are used in combination, it is possible to produce an adhesive having a high refractive index and high transparency.

Furthermore, properties such as adhesive strength and water resistance are about the same as for the adhesives of the prior art.

For suitable specific examples of N-vinyl carbzole substituent, 3-nitro-N-vinyl carbazole, 3-amino-N-vinyl carbazole, 3-methyl amino-N-vinyl carbazole, 1,2,3,4-tetrahydro-N-vinyl carbazole and 3,6-dibrom-N-vinyl carbazole can be mentioned, and for suitable specific examples of N-vinyl pyrrolidone substitutent, N-vinyl-3-methyl pyrrolidone, N-vinyl-5-methyl pyrrolidone, N-vinyl-3-benzyl pyrrolidone and N-vinyl-3,3,5-trimethyl pyrrolidone can be mentioned, for suitable specific examples of acryl amide N-substituent, N-methyl acryl amide, N-ethyl acryl amide, N-n-propyl acryl amide, N-n-butyl acryl amide, N-allyl acryl amide, N,N-

dimethyl acryl amide, N,N-diethyl acryl amide, N,N-dimethyl amino propyl acryl amide and acryloyl morpholine can be mentioned.

As a method used for curing the adhesive of the present invention, application of a high heat, electron beam, or radiation can be mentioned. Furthermore, a standard polymerization initiator is added to the adhesive of the present invention and ultraviolet or visible light is applied or heat is applied to cure the adhesive.

For the polymerization initiator used in this case, a standard thermal polymerization initiator such as benzoylperoxide (BPO) and azobisisobutylonitrile (AIBN), or standard photopolymerization initiators such as benzophenone, benzoin ether, thioxazone, benzyl, 1-hydroxy cyclohexyl phenyl ketone, hydroxy acetophenone, 1-carboxy-7,7-dimethyl bicyclo[2,2,1]heptane-2,3-dion, and camphoquinone can be used as well.

When a photopolymerization initiator is used, the curing rate can be increased significantly when a tertiary amine such as N,N-dimethyl-p-toluidine is added.

Furthermore, a redox combination where a tertiary amine and the above-mentioned organic peroxide are used in combination can be used as a polymerization initiator as well.

The mixing ratio of each component used in the adhesive of the present invention can be appropriately determined by those skilled in art according to application, etc. so that a refractive index after curing of at least 1.60 can be achieved. Thus, adjustment of the refractive index of the adhesive of the present invention can be achieved by changing the mixing ratio of compound A and compound B.

When the mixing ratio of compound A is less than 30 wt%, the refractive index (n_d) of the adhesive produced after curing drops below 1.60, at times; thus, it is not desirable.

Furthermore, in order to achieve adequate adhesion as an adhesive, it is desirable when at least approximately 2 wt% of compound B is included.

Furthermore, when the mixing ratio of compound A is 65 wt% or below, the adhesive remains a liquid at room temperature; thus, handling is easy.

Needless to say, commonly used additives such as polymerization inhibitors and ultraviolet absorbers can be included in the adhesive of the present invention. Furthermore, upon application of the adhesive of the present invention, a surface treatment such as an acid treatment, alkaline treatment, plasma treatment, or silane treatment can be applied to the optical components to further increase the adhesive strength.

In this case, the refractive index (n_d) is the refractive index when the measurement is made with the D beam of sodium (wavelength 589 nm).

In the following, the present invention is explained in further detail with application examples.

[Application Example 1]

Mixing was performed for 60 parts by weight of N-vinyl carbazole, 40 parts by weight of N-vinyl pyrrolidone, and 1 part by weight of AIBN and stirring was provided to produce a transparent adhesive of the present invention that remained liquid at room temperature. The refractive index (n_d) of the above-mentioned adhesive was 1.62. A thin coating of the above-mentioned adhesive was applied between two pieces of optical glass (Bk-7 glass) and heating was performed for 30 min in a constant temperature bath kept at 70EC. The optical glasses were firmly bonded and the cured adhesive layer exhibited a high transparency with an absence of

whitening. Furthermore, the refractive index (n_d) after curing was 1.64 and a very high refractive index was achieved.

[Application Example 2]

Mixing was performed for 60 parts by weight of N-vinyl carbazole, 40 parts by weight of N,N-dimethyl acryl amide, and 3 parts by weight of 1-hydroxy hexyl phenyl ketone and stirring was provided to produce a transparent adhesive of the present invention that remained liquid at room temperature. The refractive index (n_d) of the above-mentioned adhesive was 1.59. A thin coating of the above-mentioned adhesive was applied between two pieces of optical glass as in Application Example 1 and an intense ultraviolet of 10 mw/cm^2 was applied for 1 minute using a high-pressure mercury lamp. The optical glasses were firmly bonded and the cured adhesive layer exhibited high transparency with an absence of whitening. Furthermore, the refractive index (n_d) after curing was 1.62 and a very high refractive index was achieved.

[Application Example 3]

Mixing was performed for 35 parts by weight of 3-amino-N-vinyl carbazole, 65 parts by weight of N-vinyl pyrrolidone, 0.1 parts by weight of 1-carboxy-7,7-dimethylbicyclo[2,2,1]heptane-2,3-dion and 0.1 parts by weight of N,N-dimethylparatoluidine so as to produce an adhesive of the present invention, and the above-mentioned adhesive was applied between two pieces of optical glass as in Application Example 1 and visible light of 600 kilolux was applied for 5 minutes using a halogen lamp and curing was performed. The optical glasses were firmly bonded and the cured adhesive layer exhibited high transparency with an absence of whitening. Furthermore, the refractive index (n_d) after curing was 1.60 and a very high refractive index was achieved.

[Application Example 4]

Mixing was performed for 55 parts by weight of 3-nitro-N-vinyl carbazole, 45 parts by weight of N-methyl acryl amide, 0.1 parts by weight of camphoquinone and 0.1 parts by weight of N,N- dimethyl paratoluidine and bonding of optical glass was done as in Application Example 3. As a result, excellent adhesion and transparency were achieved, and the refractive index (n_d) after curing was 1.61.

[Application Example 5]

Mixing was performed for 60 parts by weight of N-vinyl carbazole, 40 parts by weight of N-vinyl-3-methyl pyrrolidone, 1 part by weight of BPO and stirring was performed to produce a transparent adhesive of the present invention that remains liquid at room temperature. A thin coating of the above-mentioned adhesive was applied between two pieces of optical glass (Bk-7 glass) and heat was applied for 30 minutes in a constant temperature bath kept at 70EC. As a result, the optical glasses were firmly bonded and the cured adhesive layer exhibited a high transparency with an absence of whitening. Furthermore, the refractive index (n_d) after curing was 1.63 and a very high refractive index was achieved.

Furthermore, for comparative examples, instead of compound B of the present invention, production of adhesives was carried out with (meth)acrylates such as 2-hydroxyethyl(meth)acrylate and bisphenol A glycerol-modified di(meth)acrylate commonly used for acrylic based adhesives, and parachlorostyrene.

[Comparative Example 1]

Instead of N-vinyl pyrrolidone, 2-hydroxyethylmethacrylate was used and composition was produced as in the case of Application Example 1. Uniform dissolving of N-vinyl carbazole was not possible, and it was not possible to use the composition as an optical adhesive.

[Comparative Example 2]

Mixing was performed for 50 parts by weight of N-vinyl carbazole, 50 parts by weight of parachlorostyrene, and 0.05 parts by weight of AIBN so as to produce a transparent composition that remains liquid at room temperature. The refractive index (n_d) of the composition produced above was 1.62. Heating was performed at 65EC for 3 hours and when bonding of the optical glasses was performed, whitening was observed in the adhesive layer.

[Comparative Example 3]

Mixing was performed for 100 parts by weight of bisphenol A glycerol-modified diacrylate and 3 parts by weight of 1-hydroxycyclohexylphenyl ketone and bonding was performed as in Application Example 2. The refractive index (n_d) of the cured adhesive was 1.58.

[Effect of the invention]

As shown in the results obtained in the above-mentioned application examples, the adhesive of the present invention exhibits high refractive index, good adhesive properties, and high transparency. Furthermore, as is clearly shown in the results for the comparative examples, when compound B of the present invention is omitted, an adequate dissolving of the compound A is not possible, and whitening of the adhesive is observed, or even when uniform dissolving is possible, whitening is observed after curing of the adhesive due to poor copolymeric properties with compound A, or [the composition] failed to achieve the target refractive index.